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DURABLE HEART DIAPHRAGM FROM ORDERED POLYMER FILMS

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The purpose of this Phase I SBIR study was to investigate the feasibility of using the ordered polymer poly-p-phenylene benzobisthiazole (PBZT) in high flex life applications. Applications of particular interest include biomedical applications such as the artificial heart or prosthetic heart valves. An accelerated flex life test rig based on a vibration principle was developed that was capable of subjecting film samples to over 3 million flexural cycles per 24 hr period. Film samples were prepared of neat PBZT and of PBZT in an interpenetrating network (IPN) with silicone and with polyurethane. The IPN with polyurethane was also subjected to biocompatibility screening tests.

The results demonstrated the capability of the accelerated life test rig to provide high cycle rate and adequate flexure to fatigue the film samples. The test was run for 100 million cycles. Information obtained indicated that flex life of PBZT was improved by forming an IPN with a polyurethane. Biocompatibility screening tests demonstrated

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feasibility of using the material in contact with blood.

The study indicated there is a potential for a high flex life material to be made from ordered polymers formed in an IPN with polyurethane. Since other ordered polymers of the same family have an even greater potential for high flex life, the results of this study suggest that a superior material for high flex life applications may be achieved. Aerospace potential includes use of these materials in light weight survivable fuel tanks and conduits, cryogenic fluid vessels, structural skins and a wide variety of hydraulic and pneumatic system components.

TABLE OF CONTENTS

DURABLE HEART DIAPHRAGM FROM ORDERED POLYMER FILMS

A PHASE I, SBIR FINAL RESEARCH AND DEVELOPMENT REPORT

A) PURPOSE OF THE WORK

Introduction - The ordered polymer poly-p-phenylene benzobisthiazole (PBZT) is an ordered polymer developed by the U.S. Air Force for aerospace applications. Although this ordered polymer was developed for high strength, there is reason to believe, based on its microstructure, that the material may have excellent fatigue resistance. It is also possible that the PBZT in combination with other materials, (for example in combination with polyurethane in an inter-penetrating network or IPN), might yield a superior material for high flex life applications. In aerospace applications, the thin tough films could be used for light weight survivable fuel tanks and conduits, cryogenic fluid vessels, structural skins, and a wide variety of hydraulic and pneumatic system components. There are also biomedical applications such as flexing diaphragms for the artificial heart. Additional biomedical applications could consist of replacement of the diseased natural valves of the heart and of damaged blood vessels of the body with prosthetic heart valves and prosthetic blood vessels. These biomedical applications require particularly high flex life. The purpose of this work was to determine the feasibility of PBZT films and/or PBZT IPN films to meet the demands of high flex life applications. A secondary purpose was to determine the feasibility of these films to meet the biocompatibility requirements for long-term prosthetic device implant applications.

Biomedical Applications-Background Information - For the biomedical applications, which require the very high flex life capable of withstanding 40 to 50 million heart beats per year, the standard material of choice is currently polyurethane. As reported in a recent request for proposals from the National Institutes of Health (NIH) on implantable, total artificial hearts (REF 1), the intended durability of the entire device was noted to consist of a 5 year period. As reported by the University of Utah (REF 2), over a period of several years artificial hearts had been placed on real time testing. For real-time testing of 8 Jarvik-7 artificial ventricles in water (of the best diaphragm design and fabrication methodology at the time), three failed: The longest surviving 4.75 years, another failing at 3.08 years, and one at 1.3 years. The remaining five were reported as continuing, but had not been on test long enough to determine whether they could exceed the five year criteria. Early failures have also occurred in animal experiments (less than a year) (REF 2). Thus, the ability of polyurethane to consistently meet the 5 year durability requirement is still questionable. Furthermore, in the replacement of diseased natural valves of the heart, where it is known that superior performance could be achieved from prosthetic valves that are constructed to flex like the natural valves they replace, no flexing component valves have yet been developed of a prosthetic material. Instead, mechanical valves that can last the necessary 10 to 20 years of operation have been developed that are of a significantly different design than the natural valve, and yield sub-optimal performance compared to the natural valves they replace. If a polymer with superior flex life can be made, a significant advancement in prosthetic heart valves would be possible.

From this background it is obvious that a relatively large number of flexural cycles are likely to be necessary in order to obtain feasibility data on the flexural fatigue life of the PBZT materials. Furthermore, the time period of a Phase I feasibility study is limited to 6 months, and thus necessitates the use of a custom designed, accelerated test rig. The design and fabrication of a test rig that could subject the PBZT film samples in this study to millions of cycles of flexure in a relatively short time was thus an additional goal of this study.

PBZT Film - Background Information - Three basic types of material can be used for high flex life applications:

-Unreinforced film
-Fiber reinforced (fabric) composites
-Self-reinforced ordered polymer (PBZT) films.

The polyurethane films currently in use in artificial heart diaphragms (such as Biomer and Angioflex) fall into the first category and have the advantages of fabrication ease and flexibility. However, unreinforced films are inherently weak with ultimate tensile strength in the range of 5,000 to 8,000 psi. This may be the cause of the creasing and subsequent pinhole leaks observed with these materials (REF 2). Low tensile strength also limits how thin the films can be made and used.

Fiber-reinforced composites offer a potential solution to the strength problem since high strength fibers of graphite, Kevlar, and polyethylene are readily available with ultimate tensile strengths over 400,000 psi. The fibers must be woven into a fabric, or cross-plied in order to achieve strength in all directions, and this ultimately results in abrasion and short fatigue life.

Self-reinforced ordered polymer films offer the best potential for a durable diaphragm without the drawbacks of the low tensile strength of polyurethane, nor the abrasion problems of fiber-reinforced composites.

The rod-like molecular structure of PBZT gives rise to a self-reinforced microstructure. Self-reinforced ordered polymer films can have the high tensile strength (at least 50,000 psi in all directions in the plane) associated with fiber-reinforced composites, but without the drawbacks (such as abrasive wear) of these composites. PBZT ordered polymer films can be made with homogeneity down to a very fine scale, less than 0.1 micron. For this reason, the microfibrillar network should have much less differential strain than the fiber-reinforced composites in which the fiber diameter is an immense 25 microns or greater. Thus, PBZT film could show excellent fatigue resistance along with its high strength.

Nu-Tech Industries, Inc.'s subcontractor, Foster-Miller, Inc., has processed thin high strength films from PBZT which show a self-reinforcing fibrillar microstructure. In addition to developing techniques to produce biaxially oriented films, Foster-Miller has experience in methods of impregnating the microfibrillar network with a secondary material at a point in the process when the network is open, thereby producing a PBZT IPN material. By using polyurethane as the secondary material, the proven flex life of polyurethane may be improved by the PBZT, and the proven biocompatibility of polyurethane could enhance the biocompatibility of the PBZT.

Specific Aims - The following specific aims were derived from the above background information as being appropriate in order to determine the feasibility of PBZT film of providing high flex life, and biocompatibility as an implantable material.

1. Design, build, and evaluate a flex life test rig that achieves a suitable balance between speed of testing and proper flexural excursion of test samples.

2. Test PBZT alone and PBZT IPN materials for flex life to determine the potential for PBZT to provide a more durable artificial heart diaphragm.

3. Evaluate PBZT alone and PBZT IPN materials in biocompatibility screening tests to determine their potential as blood contacting materials.

B) DESCRIPTION OF THE WORK PERFORMED

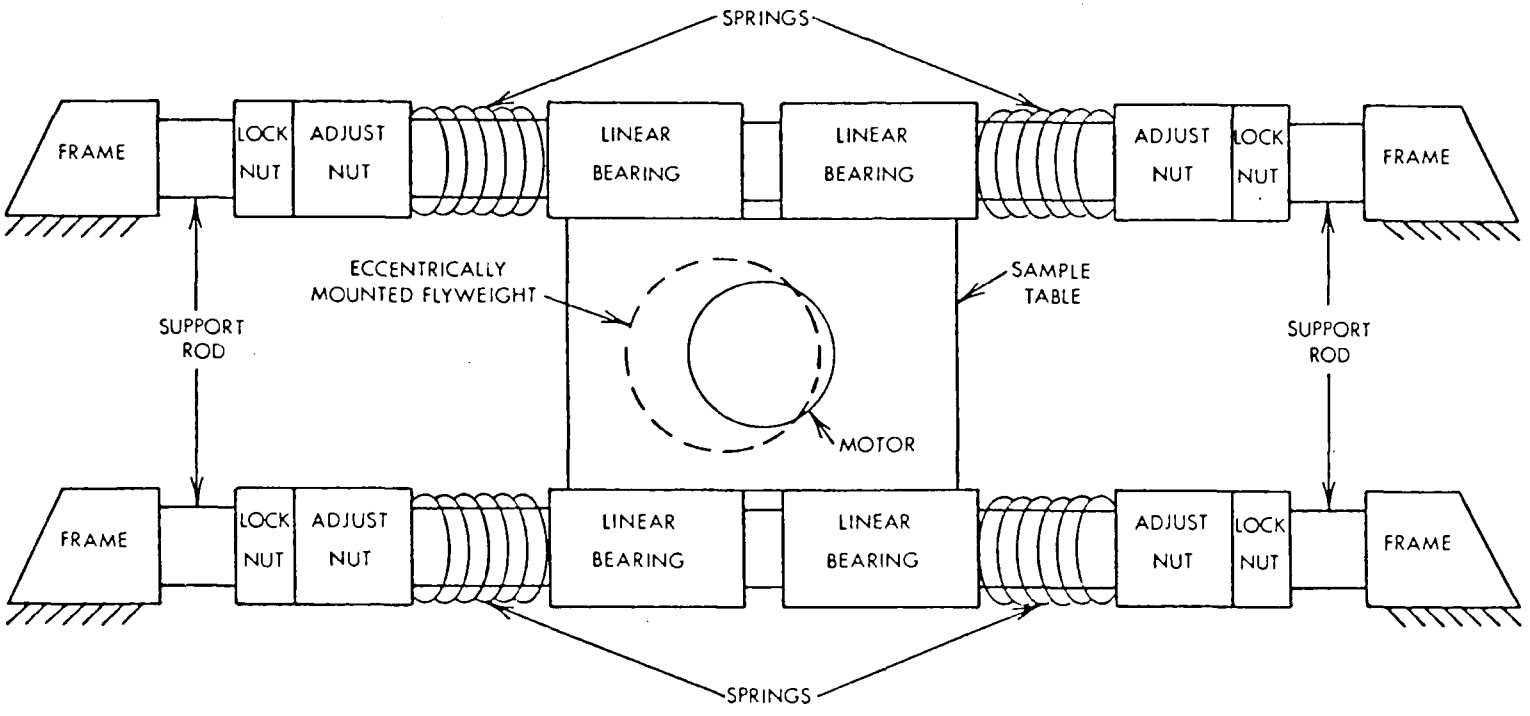
B.1 Development of a Flex Life Test Rig - In order to achieve a test rig capable of subjecting the test samples to such a large number of flexural cycles in a short period of time, we selected a testing approach based on vibration. Figure 1 shows schematically the vibration table that was developed. Our goal was to devise a method of vibrating a sample holding fixture in a linear fashion, with a relatively large amplitude. The sample table was constrained by design to move linearly. This was accomplished by mounting the table to linear bearings which in turn were designed to ride on steel rods, one on either side. In order to provide a sinusoidal forcing function to produce the vibration, a motor was mounted to the table that rotated an eccentric mass. The restoring function for the vibration was provided by four springs mounted on the steel shafts, and located between the linear bearings and the frame of the device. As the eccentric mass rotates, the sample holding table vibrates, but because the motion is constrained by the steel rods and linear bearings in one direction only, the table moves linearly.

Figure 2 is a schematic side view of the device showing the film samples hanging from the underside of the table. The samples were rectangular, and had holes punched in both ends to provide a means to clamp the samples to the sample holding bar. By clamping the samples at both ends, a loop of film hangs down and as the sample table vibrates the loop is subjected to flexural motion as shown in Figure 2. At speeds of rotation of the motor and eccentric weight between 2200 and 2400 revolutions per minute, the samples are subjected to the same rate of flexural cycling, yielding a highly accelerated flex life fatigue test rig.

B.2 Test Rig Design Rationale -

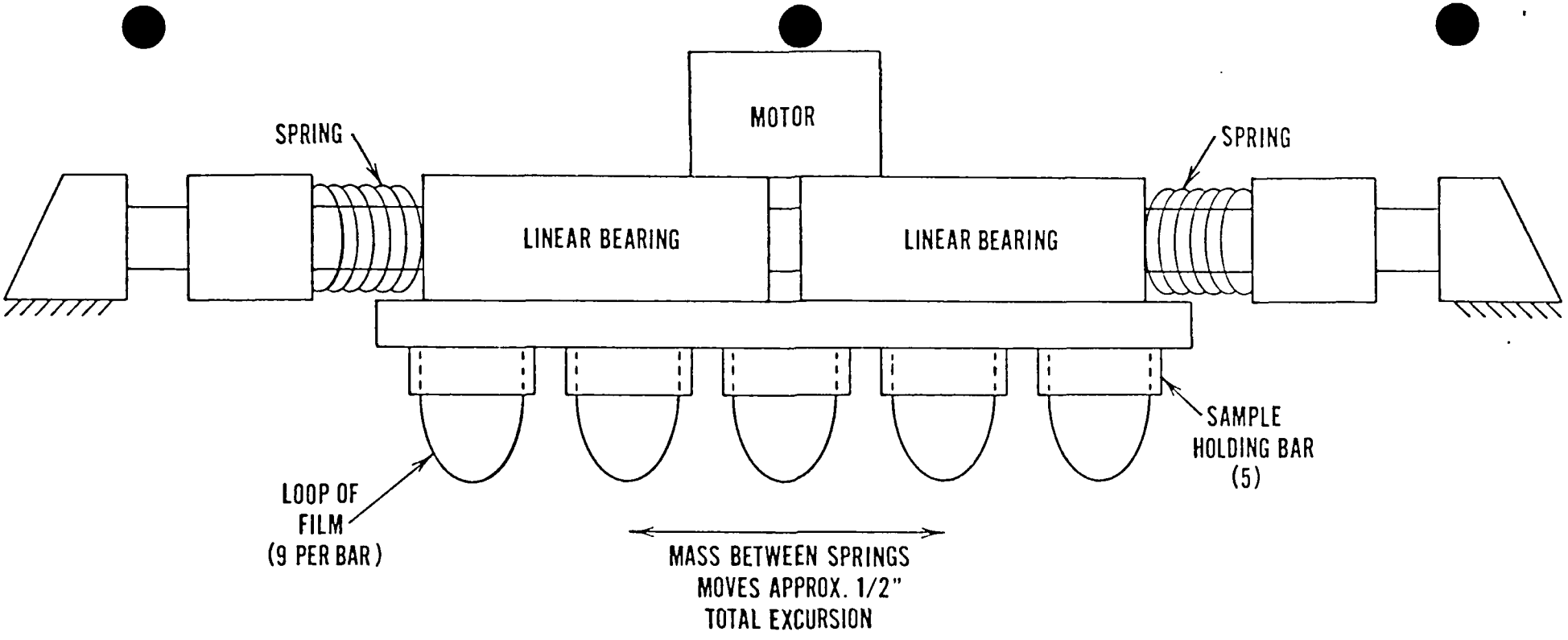
The governing equation for the vibrational motion of the test rig, assuming damping to be negligible, is as follows:

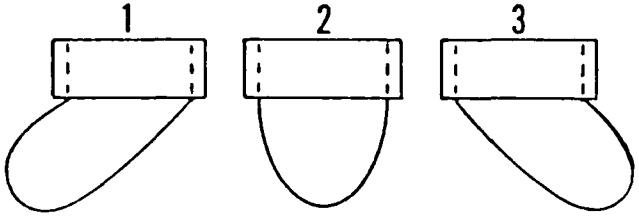
$$x_m = \frac{P_m}{K - M\omega^2} \qquad \text{Eq. 1.}$$



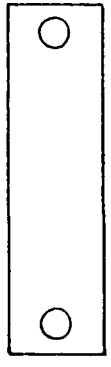
SCHEMATIC OF VIBRATION TABLE DEPICTING MAJOR COMPONENTS

Figure 1





APPROXIMATE
MOTION OF EACH
SAMPLE AS TABLE MOVES
(2200-2400 CYCLES/MINUTE)



SHAPE OF
FILM SAMPLE

Figure 2

where x_m is the amplitude of the sinusoidal vibration

P_m is the amplitude of the sinusoidal forcing function

K is the equivalent spring constant

M is the total mass of the components that are in motion

and ω is the frequency of vibration.

The forcing function, P_m, is dependent upon the mass of the eccentric weight (m), the eccentricity of the center of mass (r), and the angular velocity (ω), according to the following relationship.

$$P_m = mr\omega^2 \qquad \text{Eq. 2.}$$

From the vibration equation it is obvious that a frequency of $(K/M)^{1/2}$ results in an infinite amplitude. This frequency is known as the natural frequency. In reality, since there is some damping in the system, although the amplitude at the natural frequency obviously is not infinite, it will be the highest amplitude. Our goal in designing the fixture was to achieve a natural frequency as high as reasonably possible, in order to achieve a highly accelerated fatigue life test rig. The intent was then to operate as close to the natural frequency as would be necessary in order to achieve adequate excursion of the samples.

In order to achieve a high natural frequency the spring constant (K) must be large with respect to the moving mass (M). However, a large spring constant produces large forces on the vibrating sample table that must be met with a considerable strength of structure, which leads to increased mass. Thus, as with all designs, compromise had to be made. The compromise in this design was between spring constant selection, the resulting force on the vibrating table, and the mass required to support the force. This was essentially the design balance that had to be met in order to produce a workable fixture.

Yet another design consideration was the life of the components. The linear bearings would see considerable travel distance, the springs would be subjected to a significant number of flexural cycles, the motor and lead wires would have to withstand vibration, and the bearings supporting the rotation of the eccentric mass would be subjected to considerable forces that would be likely to cause wear and bearing failure. The design was therefore constrained further by the available components, and by the limitations placed on component selection by estimates of life of the component in the application.

As a result of multiple iterations, considering a range of available components, and estimating the moving mass, the following tentative values were determined for the vibration equation.

for the forcing function: $mr=0.007448$ lb-sec^2

for the spring constant: K=50,880 lb/ft

for the moving mass: $M=25$ lb_m

yielding a natural frequency of 2,444 cycles/min

By operating the motor above the natural frequency, at 3,000 cycles per minute the amplitude we could expect with negligible damping is therefore calculated to be +/- 0.34 inches, for a full peak to peak excursion of 0.68 inches. The amplitude was estimated to provide a reasonable angle of flexure for the loop of film attached to the sample table as depicted in Figure 2, and the 3,000 cycles per minute would yield over 4 million cycles in a 24 hour period of operation.

B3. Selection of Individual Test Rig Components -

Eccentric Mass - A sketch of the eccentric mass is shown in Figure 3. The eccentric mass was circular with a diameter of 4 inches and a thickness of 0.75 inches, made of steel (density 0.283 lb/in^3). A 3/4 inch hole was drilled in the mass to match with a 3/4 inch shaft which in turn was press fitted to the motor shaft. By locating the 3/4 inch hole with its center offset from the center of mass by 1.079 inches, the product of mass (m) and mass eccentricity (r) is 0.007448 $lb\text{-}sec^2$. As noted above, the forcing function was determined by iteration with design criteria that considered cycling speed, amplitude of excursion, forces on the structure, and life of the components.

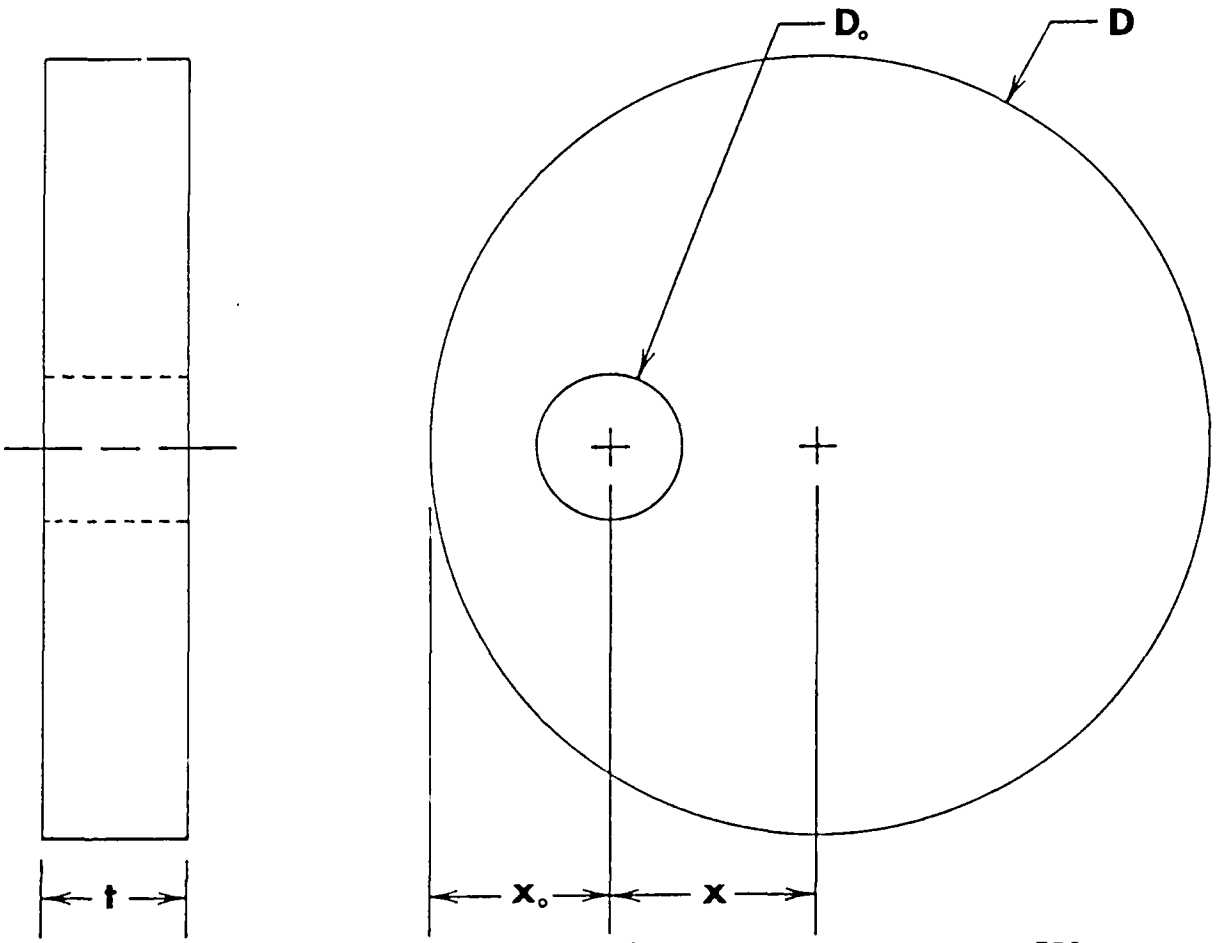
Linear Bearings - (Thomson Industries) Super Pillow Block linear bearings were selected, based on the travel life for the load that they had to carry. In order to aid in proper selection, the supplier makes available a chart depicting travel life in millions of inches as a function of the load correction factor, K_L, defined as follows when the shaft moving within the bearing has a shaft hardness of 60C Rockwell.

$$K_L = \frac{\text{Load capacity req'd}}{\text{Rolling Load Rating}} \qquad \text{Eq. 3.}$$

where the Load Capacity in our case is $P_m/4$ (load shared by four linear bearings) and the Rolling Load Rating is dependent upon the bearing selected, and generally increases with shaft diameter and bearing size.

We selected an SPB-20 bearing with a 1.25 inch shaft diameter, a length of 3 5/8 inches, a weight of 2.5 lbs and a rolling load rating of 1170 lbs. For the P_m selected, this yielded K_L of 0.157, which in turn yielded an expected life of 440 million inches of travel. It should be noted that travel life increases as K_L decreases, and thus the selection of four bearings to distribute the load, coupled with the selection of a relatively large diameter shaft in order to obtain a large rolling load rating were major factors in the selection process. However, the mass added to the moving mass also increases as load rating increases, and thus we had to balance the load carrying capability and life against the added mass on the table. To translate travel life into cycle life, it is noted that each full cycle consists of four times the calculated amplitude of the vibration. Although we calculated that the amplitude at 3000 rpm would be 0.34 inches, at this point in the design process it was decided to provide for the possibility that the amplitude could be as high as 0.5 inch since we might end up operating at lower speed and thus closer to the natural frequency. The 440 million inches thus results in 220 million cycles of life, or the equivalent of four to five years of artificial heart rate cycling.

FLYWEIGHT (STEEL)



t = .750 ± .001

X₀ = .920 ± .001

X = 1.079

D₀ = .750

D = 3.998 ± .001

Figure 3

Needle Bearing Selection - Torrington model B-128 needle bearings were selected as the bearings to support the rotating eccentric mass. By using two such bearings the load was distributed, and the expected life of the bearings was extended.

The life factor for the bearings (LF) is given by the manufacturer as a function of the basic dynamic load capacity (BDC) rating, the load (in this case equal to $P_m/2$), and the speed factor (SF) as follows.

$$LF = BDC/(Load \times SF) \qquad \text{Eq. 4.}$$

For the bearing selected, the BDC=2020, SF at 3,000 rpm was 3.88, and the $P_m/2$ was calculated at 367.5 lbs, yielding LF=1.416. From a manufacturer supplied chart the LF factor predicted 1600 hours of life. At 3000 cycles per minute the life in total cycles is thus 288 million cycles. If the speed had been selected at a higher value, the load would also have been greater, requiring a bearing selection that was longer and/or larger in diameter, features that lead to greater mass of the table.

Motor Selection - In order to select the motor, the operating torque had to be esti .ted. When the vibrating table has reached a steady state operating point, the motion of the table is such that energy is transferred back and forth between kinetic energy of motion of the table and potential energy of stored in the springs. The frictional losses in the linear bearings and in the needle bearings is where the power of the motor is spent.

The torque lost to friction in the needle bearings can be expressed as:

$$T_{LOSS} = \mu_{N.B.} P_m R_{N.B.} \qquad \text{Eq. 5.}$$

where T_{LOSS} = Torque lost to friction in the needle bearings

$\mu_{N.B.}$ = coefficient of friction for the needle bearings

$R_{N.B.}$ = Pitch Radius of needle bearings, 0.4375 in

and P_m is defined by Eq. 2.

For the linear bearings, the force of friction (F_f) is in the direction of motion of vibration, and vibration theory equates the frictional force to the product of the damping coefficient (c) and the velocity (dx/dt). The friction force is also equal to the product of the coefficient of friction for the linear bearing ($\mu_{L.B.}$) and the normal load on the linear bearings which is sinusoidal and has an amplitude of P_m (defined by Eq. 2.).

$$F_f = c(dx/dt) = \mu_{L.B.} P_m \sin\omega t \qquad \text{Eq. 6.}$$

where the displacement (x) is a sinusoidal function given by:

$$x = x_m \sin\omega t \qquad \text{Eq. 7.}$$

and where x_m is the amplitude, ω is angular velocity, and t is time.

The most convenient form of Eq. 6 is one in which the average force is expressed as a function of average velocity and average normal load, and we take for these two averages the root mean square (RMS) values:

$$(dx/dt)_{RMS} = (x_m \omega)/\sqrt{2} \qquad \text{Eq. 8.}$$

and $$(P_m \sin\omega t)_{RMS} = P_m/\sqrt{2} \qquad \text{Eq. 9.}$$

We may now rewrite Eq. 6 in terms of the average force of friction, and we note that (c) can now be calculated from the manufacturer's value for the coefficient of friction.:

$$(F_f)_{AVG} = (c\ x_m \omega)/\sqrt{2} = (\mu_{L.B.}\ P_m)/\sqrt{2} \qquad \text{Eq. 10.}$$

We must now relate the power delivered by the motor to the frictional power losses in the direction of vibration. However, we first note that the motor torque delivered to the eccentric mass is a net torque (T_{NET}) due to the torque lost in the needle bearings, and thus the torque developed by the motor, (T_{MOTOR}) can be related to the net torque and torque loss by:

$$T_{NET} = T_{MOTOR} - T_{LOSS} \qquad \text{Eq. 11.}$$

The net power delivered to the eccentric mass, and that is ultimately lost in the frictional losses in the direction of vibration is then:

$$\text{POWER} = (T_{NET})(\omega) \qquad \text{Eq. 12.}$$

The net power lost to friction can also be related to the frictional losses in the direction of vibration by integrating the frictional force in Eq. 6 with respect to displacement (x) over a cycle, and dividing by the time for a cycle. (This integration thus produces the product of force and distance which is work or energy, and then by dividing by the time we obtain power.) The result of this integration and division by the time per cycle yields:

$$\text{POWER} = (0.5)\ (c)\ (x_m)^2(\omega)^2 = (T_{NET})(\omega) \qquad \text{Eq. 13.}$$

Equations 5, 10, 11, and 13 can now be solved, given the coefficients of friction for the linear bearings and needle bearings, for T_{MOTOR}. The manufacturer's catalog for the needle bearing gives a coefficient of friction of 0.0025, and the catalog for the linear bearings gives a coefficient of friction of 0.0027. By using a multiple of 2.5 times these coefficients we introduced a safety factor into our calculations. The resulting value thus calculated for T_{MOTOR} was 46 oz-in of torque. The performance curve for the motor selected is shown in Figure 4. The expected efficiency at the operating point was deemed to be an acceptable trade-off compared to the added weight that would be required had we selected a larger motor.

Spring Selection - The criteria for selection of the springs included: (a) a hole diameter large enough to permit the support rod to pass within, (b) spring constant sufficiently high enough to yield the desired natural frequency as discussed above, (c) a deflection to free length ratio low enough to permit a projection of long life. With these criteria in mind, Danly medium-high load springs were selected. These springs are of valve spring quality, are vacuum degassed and are made of chrome vanadium for long life. Catalog number 9-4018-21 were selected with a spring constant equal to 1/4 the equivalent

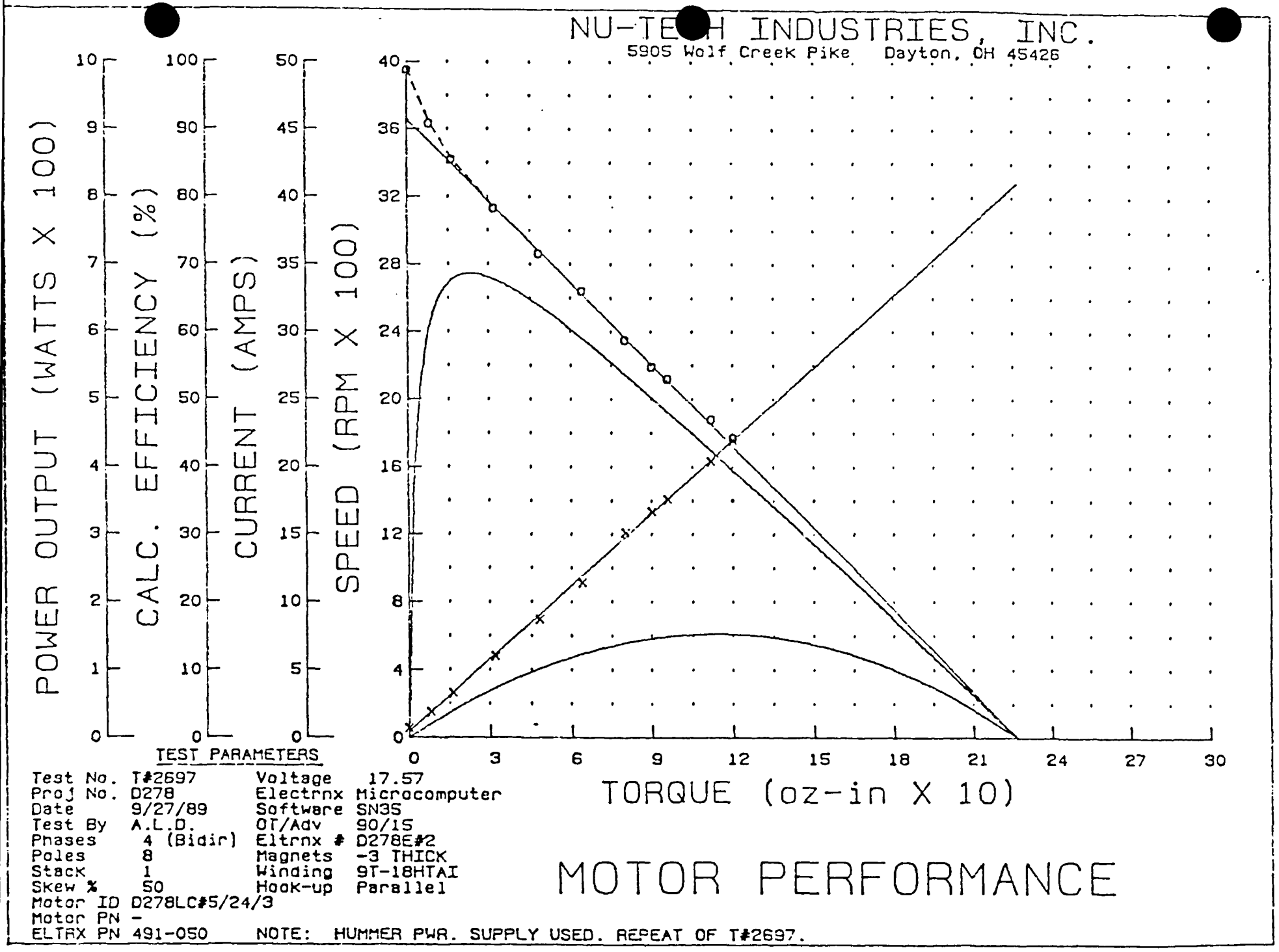

NU-TECH INDUSTRIES, INC.
5905 Wolf Creek Pike Dayton, OH 45426
POWER OUTPUT (WATTS X 100)
CALC. EFFICIENCY (%)
CURRENT (AMPS)
SPEED (RPM X 100)
TORQUE (oz-in X 10)
TEST PARAMETERS
Test No. T#2697
Proj No. D278
Date 9/27/89
Test By A.L.D.
Phases 4 (Bidir)
Poles 8
Stack 1
Skew % 50
Motor ID D278LC#5/24/3
Motor PN -
ELTRX PN 491-050
Voltage 17.57
Electrnx Microcomputer
Software SN3S
OT/Adv 90/15
Eltrnx # D278E#2
Magnets -3 THICK
Winding 9T-18HTAI
Hook-up Parallel
NOTE: HUMMER PWR. SUPPLY USED. REPEAT OF T#2697.
MOTOR PERFORMANCE

Figure 4

spring constant of 50,880 lb/ft (with the springs preloaded the equivalent spring constant is four times the spring constant of each spring for the configuration selected). The free length of these springs is 4.5 inches, and thus the expected deflection of x_m, including the preload deflection resulted in a small enough ratio of deflection to free length to warrant our expectation of long life. These springs could accommodate a shaft diameter of up to 1.5 inches, thereby accommodating the selected 1.25 inch shaft diameter.

B.4 Preparation of PBZT Samples -

Most of the effort in preparing PBZT samples focused on the processing techniques to infiltrate PBZT with polyurethane in an interpenetrating network (IPN). Film samples were also provided to Nu-Tech that consisted of PBZT neat, but since the processing techniques for biaxially oriented film were known prior to this investigation, the PBZT sample preparation did not constitute a major portion of the effort. All of the techniques described in this section were performed under subcontract by Foster-Miller, Inc. The text of this section is reproduced, with only minor editing, from a final report of the subcontractor's effort, written by Lucy Elandjian of Foster-Miller.

For the polyurethane used in the IPN, Foster-Miller selected a specific polyurethane, Vibrathane, to demonstrate the microcomposite film approach on this Phase I program. The selection of this secondary material was an involved process. The candidate materials had to meet certain requirements to qualify for this application. These requirements were high flex life (to replicate 40 to 50 million heartbeats per year), biocompatibility, low modulus ($\leq$ 1800 psi) and hardness (<70A Shore Hardness), high elongation (>300 percent) and low molecular weight prior to polymerization. Another criterion used for the selection was the availability of the material in the desired prepolymer form with a molecular weight of 1,000 or less. Due to the nature of PBZT structure and Foster-Miller processing methods, only small polymers can infiltrate inside where they are cured (polymerized and cross-linked) in situ with the PBZT film, thus forming a microcomposite.

The materials considered for IPN with PBZT film were polyurethaneureas, polyurethanes and silicones. All of these systems have, to date, been used in the development of artificial internal organs. The most successful of them have been the polyurethaneureas. Produced under trade names as "Biomer" (Ethicon Corp.), "Angioflex" (Abiomed, Inc.), "Cardiomat-610" (Contra, Inc.), "TLC-15/TLC-16 and PBS-215M" (Thoratec, Inc., Mercor Div.), polyurethaneureas have low modulus and hardness ($\leq$ 1800 psi and <70A Shore), and low hysteresis/creep properties. Their excellent biocompatibility and flexural properties make them ideal for blood pump applications.

Polyurethanes, such as "Vibrathane" (Uniroyal Chemical), "Pellethane" (Dow Chemical) and "Avcothane-51" (Avco Polymers Div.), were the predecessors to polyurethaneureas. They exhibit moderate toughness and modulus, greater than 70A Shore hardness, high hysteresis/creep and good flex life. However, the critical properties (biocompatibility and flex life) are inferior to those of the polyurethaneureas; and therefore, their use in artificial devices is now limited.

Silicones, such as "Silastic MDX-44210" (Dow Corning) and "Petrarch SS" (Petrarch System, Inc.) have low flex life, low modulus, moderate toughness and hardness. They lack biocompatibility and thus are no longer used in artificial

devices except as part of polyurethane formulations (i.e., Avcothane-51).

Problems of high molecular weight and unavailability of the prepolymer were encountered while researching the IPN material. The most appropriate materials for our application, polyurethaneureas, were originally being sold as ordinary resins. However, they were taken off the market after the discovery of their biocompatibility properties. They are currently being used as proprietary materials in the manufacture of medical devices. With the exception of Angioflex, none of these materials are available in the prepolymer form.

Under the circumstances, polyurethanes were determined to be the most appropriate material. Their critical properties were close to polyurethaneureas and they were available in low molecular weight, two-component prepolymer form. Thus, Vibrathane B670, a polyurethane elastomer, was chosen as the secondary IPN material. Vibrathane has a molecular (prepolymer) weight of 750 (cured polymer weight of 100,000) and a modulus of 2,300 psi (low enough, considering TLC-15 is 1,800 psi and Biomer 1,400 psi). Although this material is not biocompatible, it is approved for food contact.

After demonstrating the feasibility of the IPN system and Vibrathane, it may be possible to draw up an agreement with Abiomed, Inc. and use Angioflex in future investigations.

PBZT is an aromatic heterocyclic polymer which forms extended chain rigid-rod molecules. It is necessary to process PBZT while in a solution of polyphosphoric acid, for it cannot be thermoformed. The stiff rod-like molecules give rise to a self-reinforced material which has the strength of advanced fiber-reinforced composites (Figure 5), but without the drawbacks of fiber-matrix mismatch and relatively high interfacial stress. PBZT films were fabricated at Foster-Miller with a proprietary extrusion process capable of producing thin, (0.2 mil dry, neat) blown films with controllable microfibril orientation at $\pm \theta$ direction, where θ is the angle between the fiber and the machine direction. θ is tailorable for each desired application (Figure 6). The artificial heart diaphragm requires equal strength in all directions in the film plane, and for this isotropic application $\theta = \pm 45$ deg.

Biaxially extruded PBZT blown film was washed in a water bath to remove residual solvent. The film was then cut into 3-ft lengths and remained in the water bath until use. The neat PBZT films were dried at elevated temperatures and pressures to consolidate the microfibrillar structure.

Vibrathane was infiltrated into PBZT films by infusing low molecular weight oligomers into solvent-swollen films prior to drying. We estimate that the space between bundles of microfibrils, where polyurethane is to infiltrate, is less than 0.1μ. Vibrathanes's molecular weight of 750 was low enough to complete infiltration. Water-swollen PBZT films were subjected to a solvent exchange process to maintain the open microporous structure while replacing the water with acetone. The wet tubes were soaked in a 20 percent solution of polyurethane in acetone, then dried under controlled stress, making a microcomposite film of PBZT and polyurethane.

Thick (1.2 mil dry, neat) biaxial $\pm$ 45 deg films were extruded along with thin films. Thicker film was easier to process at uniform thickness under the given conditions with existing equipment. It was possible to process thin

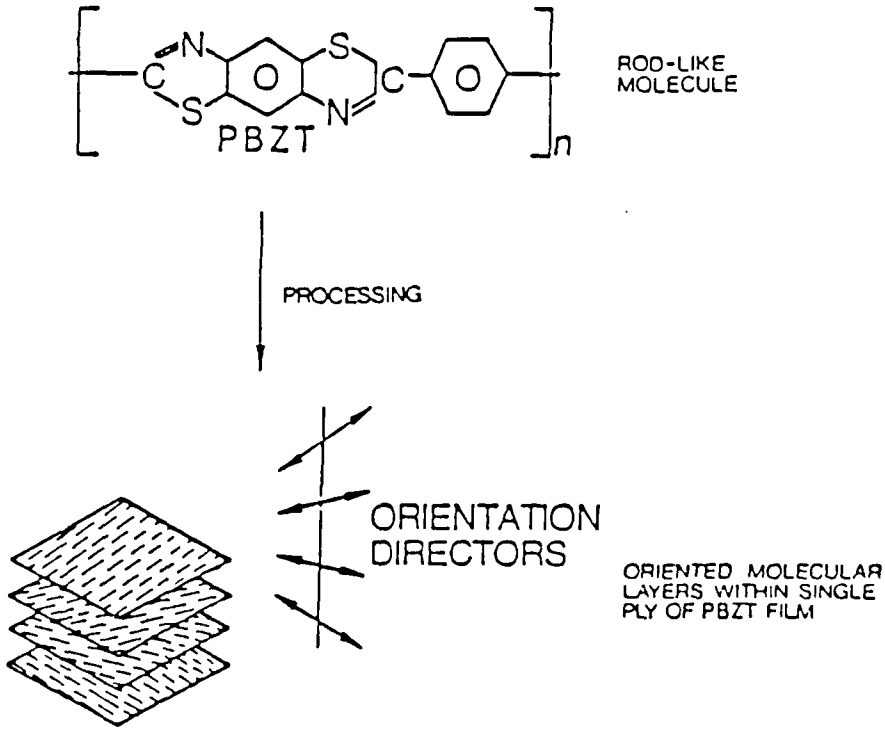


Figure 5. Rod-Like Molecules Give Rise to Self-Reinforced Microstructure Within Single Plies of PBZT Film

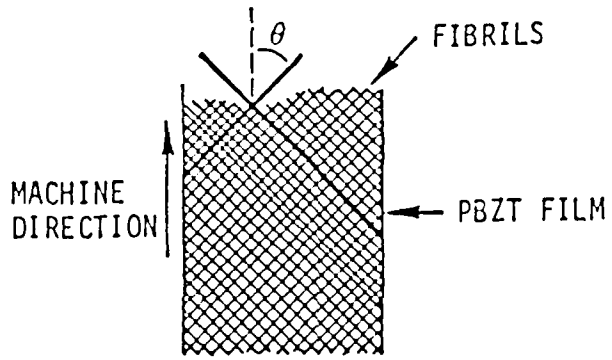


Figure 6. PBZT Fibrillar Network Direction

biaxial film; however, there was an approximate 20 percent deviation in thickness. The die could be redesigned to maintain uniformity throughout the film in subsequent programs. Such a task was not feasible with the Phase I funding; nevertheless, the thin films produced to date are adequate for this study, and show promising future performance.

Thick film was also interpenetrated with Vibrathane.

For better control over the experiment, infiltration with a silicone elastomer was completed. The results of the experiment should provide a good database for subsequent investigations. The silicone was interpenetrated using the same solvent exchange process as the polyurethane, except 30 percent dimethyl dimethoxy silane (molecular weight = 240) was used in the last step. After a 4-hr imbibition period, 20 ml of water and 10 drops hydrochloric acid were added to the solution to catalyze the formation of a silicone. Figure 7 describes the reaction. The PBZT film was allowed to remain in this solution for 4 more hours. The film was then dried under the same conditions as the PBZT/polyurethane film.

The following film samples were delivered to Nu-Tech:

a) six (6) sheets of PBZT neat @ 8 in X 36 in X 0.2 mil

b) one (1) sheet of PBZT neat @ 8 in X 20 in X 1.2 mil

c) five (5) sheets of PBZT/Vibrathane @ 8 in X 36 in X 1.3 mil

d) one (1) sheet of PBZT/Vibrathane @ 7 in X 18 in X 3.3 mil

e) one (1) sheet of PBZT/Silicone @ 8 in X 36 in X 0.4 mil.

B.5 Biocompatibility Testing - Nu-Tech conducted biocompatibility tests by sending samples of PBZT alone and PBZT combined with polyurethane (Vibrathane) in an Interpenetrating Network to the North American Science Associates, Inc. (NAMSA). NAMSA is a biomedical laboratory complex specializing in the biological safety testing of medical devices. The laboratories are registered with the Food and Drug Administration (FDA) and their testing services are used by numerous companies world wide to satisfy applicable regulations for regulatory agencies including the FDA. For an artificial heart diaphragm material, NAMSA specified four suitable tests for the Phase I effort.

These tests determine not only the fundamental biocompatibility, but are also capable of identifying substances that can be toxic, yet are introduced in the fabrication process and that can be subsequently removed.

Two of the tests recommended by NAMSA are classified as qualification tests and are designed to characterize the material chemically (A USP Physico Chemical test), and biologically (A Media MEM extract test). In the latter, cell cultures are exposed to an extract of the material, and thus any toxic effect from potential leachable substances is readily determined. This is one of the most sensitive cell toxicity tests that can be performed, and thus is an excellent screening test for new materials.

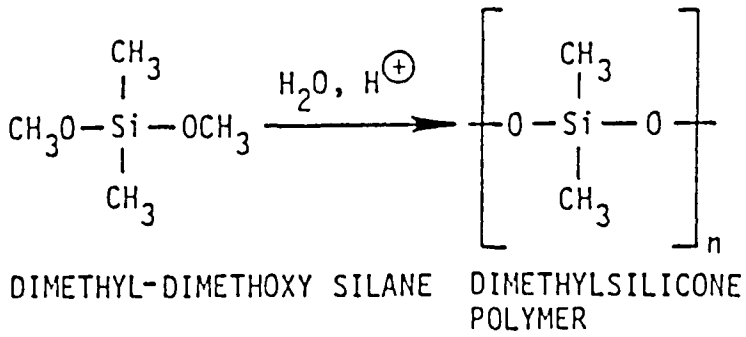


Figure 7. Hydrolysis of DMDMOS to Silicone Elastomer

The Physicochemical test is a battery of four tests used to characterize and compare new polymeric materials or to audit materials used in production. The tests provide basic information about the presence and nature of water-soluble extractables and residues, particularly those that may cause chemical toxicity problems. A 606 sq. cm portion of each of the test materials was extracted at 70 degrees C for 24 hours in 101 ml of USP Purified Water. Non-volatile reside, residue on ignition, heavy metals as lead and buffering capacity were determined in the eluate as outlined below.:

Buffering Capacity - a 20 ml aliquot of sample was titrated to pH 7.0 by 0.01 normal acid or base. The result was expressed as the volume in ml required to reach pH 7.0. A maximum of 10 ml titrant is allowed.

Heavy Metals as Lead - A color comparison was made between an aliquot of the deionized water eluate and an aliquot of a blank containing 1 ppm of lead. The sample passes if the eluate does not contain more than 1 ppm of metals as lead.

Non-Volatile Residue - a 50 ml aliquot of the eluate was evaporated to dryness and the weight of the residue was determined. The differences between the sample and the blank cannot exceed 15 mg.

Residue on ignition - The residue from the non-volatile residue test was charred, then ignited in the presence of sulfuric acid at 800 degrees C. The difference between the amounts of residue on ignition was obtained for the sample and blank and cannot exceed 5 mg.

The Cytotoxicity-MEM Elution test or cell culture test is a rapid, economical, in vitro approach to determining biocompatibility of material intended for use in medical devices. The mammalian cell culture systems employed are sensitive and can therefore be utilized in screening programs as well as in quality control and audit programs. Because of the great sensitivity of these procedures, results which indicate that a material is cytotoxic must be viewed in conjunction with results of applicable in vivo studies and in light of the intended use of the product. Cell culture models represent practical, useful in vitro alternatives to animal research.

Four cell lines are used at NAMSA: L-929 mouse fibroblast, WI-38 human embryonic lung, MRC-5 human embryonic lung, and rabbit corneal cells. The L-929 cell line was suggested for our tests because it is easiest to culture, the supply is unlimited and it is very dependable. NAMSA has shown excellent agreement among all four cell types tested against materials of low, intermediate and high degrees of cytotoxicity.

A monolayer of L-929 Mouse Fibroblast cells was grown to confluency and exposed to an extract of the test samples prepared by placing the test articles in 20 ml of Minimum Essential Medium (Eagle) and bovine serum (5%) and extracting at 37 degrees C for 24 hours. An MEM aliquot was used as a negative control. A Positive Control was also used that was toxic at a dilution of 1:2 at 24 hours. After exposure to the extract, the cells were examined microscopically for cytotoxic effect (CTE). Presence (+) or absence (-) of a confluent monolayer, intracellular granulation, cellular swelling and crenation and the percentage of cellular lysis were recorded. CTE was scored as either Nontoxic (N), Intermediate (I) or Toxic(T) as follows:

N = Indicates a negative or nontoxic response.

I = Indicates an intermediate response, a subjective assessment of the extent of cellular response.

T = Indicates a positive or toxic response consisting of greater than 50% cell death.

The remaining two tests are in NAMSA's category of advanced biocompatibility tests. An In vitro Hemolysis-Extract test was performed in order to determine any potential of the materials to cause red blood cell lysis by chemical leachables. This test is typically used in the screening of a variety of biomaterials and polymers intended to contact blood. The second test is a Muscle Implantation test to detect macroscopic or microscopic tissue reactions. The tissue reaction is evaluated by a pathologist and compared to the USP (United States Pharmacopeia) control plastic similarly implanted in the same test animal.

The In vitro Hemolysis Extract Test placed a 180 cm portion of each test material in 30 ml of 0.9% sodium chloride solution and extracted at 121 degrees C for 1 hour. The extract was divided into two tubes of 10 ml each and allowed to cool to room temperature. To each extract and similarly treated control tube was added 0.2 ml of rabbit blood previously collected in a vacuum tube containing E.D.T.A. The tubes were inverted gently to mix the contents, then placed in a constant temperature water bath at 37 degrees C for 1 hour. The blood-saline mixture, and positive and negative controls were then centrifuged for 10 minutes at a speed of not less than 2100 RPM. The absorbence of each sample solution was determined spectrophotometrically at 545 nm. Similarly, absorbances were recorded for the positive control (10 ml water and 0.2 ml blood) and the negative control (10 ml 0.9% sodium chloride solution and 0.2 ml blood.)

Muscle Implantation Test - Two healthy adult New Zealand White rabbits weighing not less than 2.5 kg. were used as test animals. The rabbits were housed individually and allowed food and water ad libitum. Prior to the implantation, the back of each animal was clipped on both sides of the spinal column. All loose hair was removed after clipping and prior to implantation to prevent entry into the implantation site.

Four strips (minimum) of steam sterilized test material, approximately 1 mm wide and 10 mm long were introduced into the right paravertebral muscle of each rabbit. Two strips of U.S.P. negative control plastic were implanted in the left paravertebral muscle of each rabbit.

The animals were humanely killed 14 days after implantation and the entire paravertebral muscle on each side of the spinal column removed. Cross sections of the muscles were made to locate the implants. The tissue surrounding the center portion of each implant was examined macroscopically and microscopically by a Pathologist for any reaction. Tissues to be examined microscopically were preserved in 10% Neutral Buffered Formalin, sectioned and stained with Hematoxylin and Eosin.

B.6 Film Sample Test Preparations and Examination Procedures - In order to cut the film samples from the large sheets supplied by Foster-Miller the sheets were first examined to determine regions that appeared to be of uniform thickness. The film was laid on a flat, clean and dry surface. The material was smoothed out to insure that there were no wrinkles or folds. An exacto knife was used with a new number 11 blade and a steel straight edge was used as a guide. The PBZT materials were cut slightly longer than the control Angioflex polyurethaneurea films because the mounting procedure consisted of the Angioflex material used as a base or underlayment material. A hole punch was used to make a clean hole in the ends where they were subsequently attached and clamped to the sample holder of the vibrating table.

On the sample table, five rows or five sample bars each contained nine mounting locations. Nine samples of each of the five PBZT materials were placed on the table, and each had its own underlayment of the control Angioflex material. This in tandem mounting with the Angioflex material was necessary, particularly for the thin materials, because of the tendency for static electricity to curl the material and interfere with consistent flexural motion. The Angioflex material thus provided for a reasonable flexural consistency in spite of the differences in thickness of the PBZT materials. However, our observations under a strobe light indicated that the thinner materials contained additional nodes of flexure. Our intent, however, was to concentrate on comparing the point of common flexural motion between the sample types; i.e. the region near each clamped end of the samples. In locating five different materials on the table the order of placement was varied in order to evenly distribute the material types across the five sample bars. In this manner we sought to eliminate any influence of location on the sample table on the outcome of the test.

A sample of the Fatigue Test Inspection Sheet is included on the next page. Pertinent identification material such as material type, and location of the material on the sample table were recorded. The materials were all subjected to a preliminary examination under 20 power with a stereoscopic microscope. Comments were recorded for each sample, and any noteworthy defects were noted on a drawing of the test sample for future reference. This preliminary examination revealed that the PBZT samples of all types typically included a wrinkled appearance, and numerous regions that seemed to contain areas that gave the appearance of "pin holes". Occasionally an edge was found to have a fuzzy fibrous appearance, and these locations were also noted. The materials were not visually homogeneous, and the wrinkled appearance was in sharp contrast to the very smooth and homogeneous appearance of the Angioflex.

NU-TECH INDUSTRIES, INC.
DAYTON, OHIO

FATIGUE TEST INSPECTION SHEET

SAMPLE DATA

TEST NO _____ DATE _____
MATERIAL _____
LOT NO _____
SUPPLIER _____
SAMPLE NO _____
COMMENTS _____

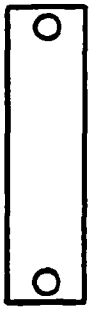
TEST PARAMETERS

MOTOR/ROTOR _____
ELECTRONICS _____
SOFTWARE _____
MOTOR SPEED _____
SAMPLE LOCATION _____
COMMENTS _____

INSPECTION #

DATE _____
RUN TIME _____ HRS _____ MINS
CYCLES _____
TYPE

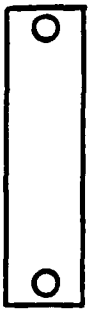
OBSERVATIONS



INSPECTION #

DATE _____
RUN TIME _____ HRS _____ MINS
CYCLES _____
TYPE

OBSERVATIONS



INSPECTION #

DATE _____
RUN TIME _____ HRS _____ MINS
CYCLES _____
TYPE

OBSERVATIONS

INSPECTION #

DATE _____
RUN TIME _____ HRS _____ MINS
CYCLES _____
TYPE

OBSERVATIONS

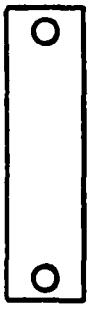


Figure 8

C) RESULTS

C.1. Test Rig Performance Results -

Vibrational Performance - The operation of the vibrational table was examined over a speed range near the desired speed of rotation of the motor in order to determine the relationship between amplitude and vibration. Speed was monitored electronically through the motor controller. A piece of thin cardboard was clamped to the fixed frame support of the table and allowed to contact a pen attached to the moving portion of the table. In this manner the length of the pen marking on the cardboard could be measured to give peak to peak amplitude (i.e $2 x_m$). Figure 9 is a plot of peak to peak amplitude vs speed of rotation. The data reflects the typical steep rise in amplitude as the vibrational frequency is increased toward the natural frequency.

By observing the vibrational motion of the table with a strobe light, it was immediately obvious that the frame also moved with the same vibrational frequency, and that we had to take into account that the amplitude as measured above was the motion relative to the moving frame. The actual motion relative to a fixed reference was approximately 80 percent of the amplitude plotted in Figure 9. The motion relative to the fixed reference frame is the true amplitude of vibration. We also observed that the spring motion consisted of compression from both ends (i.e. from the table end of each spring, and also from the frame end of each spring). This resulted in a vibrational node located at approximately one coil from the frame end (out of 4.5 coils). The effective spring length acting on the table is thus reduced, and we would expect that the effective spring constant would in turn be significantly increased.

Using Eq. 1, for the measured amplitude (relative to a fixed reference) and for the measured moving mass of 27 lbs the effective spring constant is calculated to be 72,719 lb/ft, compared to the spring constant of 50,880 lb/ft that was expected. This increased value thus correlates with the observation of reduced effective spring length. By using this larger value of spring constant and the measured mass the calculated natural frequency is 2,811 cycles per minute (compared to the design calculation of 2,444 cycles per minute. Thus, we found that for the operation range plotted in Figure 9 we are operating below the natural frequency.

By monitoring steady state temperature of the motor, we found that operation at approximately 2,400 RPM provided for acceptable motor temperature. For further safety and long term reliability of the motor four small fans were placed beside the vibration table, directed at the motor, to maintain the motor temperature as cool as possible. As the experiment progressed, and as motor design was altered (discussed below), slight drops in efficiency warranted decreasing the operating speed to as low as 2200 rpm, but observations of film sample movement indicated that even though amplitude of motion of the table was slightly decreased, the actual motion of the film samples did not appear to be compromised.

Monitoring the operating torque by comparing current during operation with current-torque characteristics determined by motor characterization tests, the torque at 2400 RPM was approximately 90 oz-in compared to the 46 oz-in that we had estimated during the design phase. This meant that operation was at a lower efficiency than expected. Higher efficiency at the operating point would

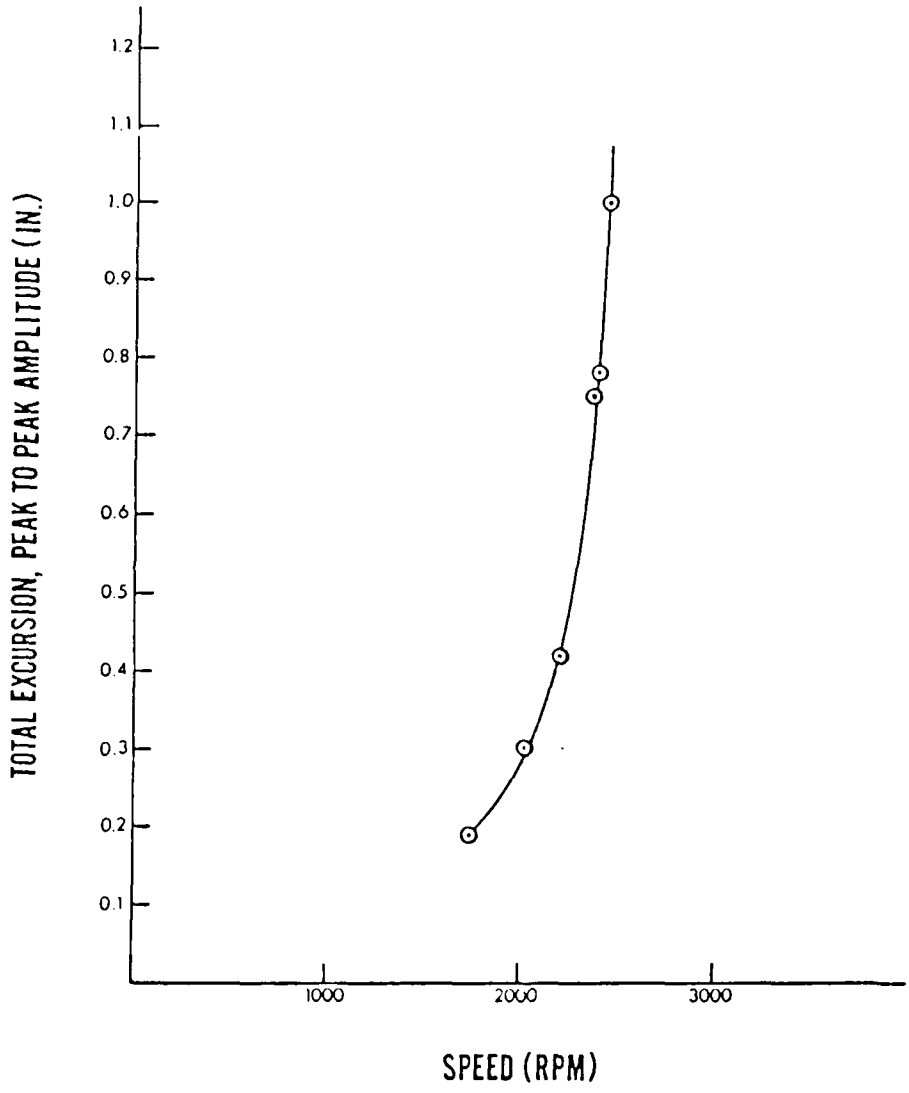

TOTAL EXCURSION, PEAK TO PEAK AMPLITUDE (IN.)
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
1000
2000
3000
SPEED (RPM)

Figure 9

have permitted faster operation with even greater amplitudes. The cause for the increased torque is assumed to be additional friction that was not accounted for. The likely cause of the increased friction was rubbing between the ends of the springs and the brass retainers. Future designs might use some type of bearing arrangement at this location to cut down on the friction. Friction was high enough at this location to cause fine particles of brass to be continually eroded.

In conclusion, although we did not anticipate the significant motion of the frame, nor the added friction at the spring retainers, the final result was acceptable in that 2,400 cycles per minute still resulted in a high rate of cycling. Furthermore, the amplitude of oscillation was significant as judged by observation of the motion of the loops of film under a strobe light.

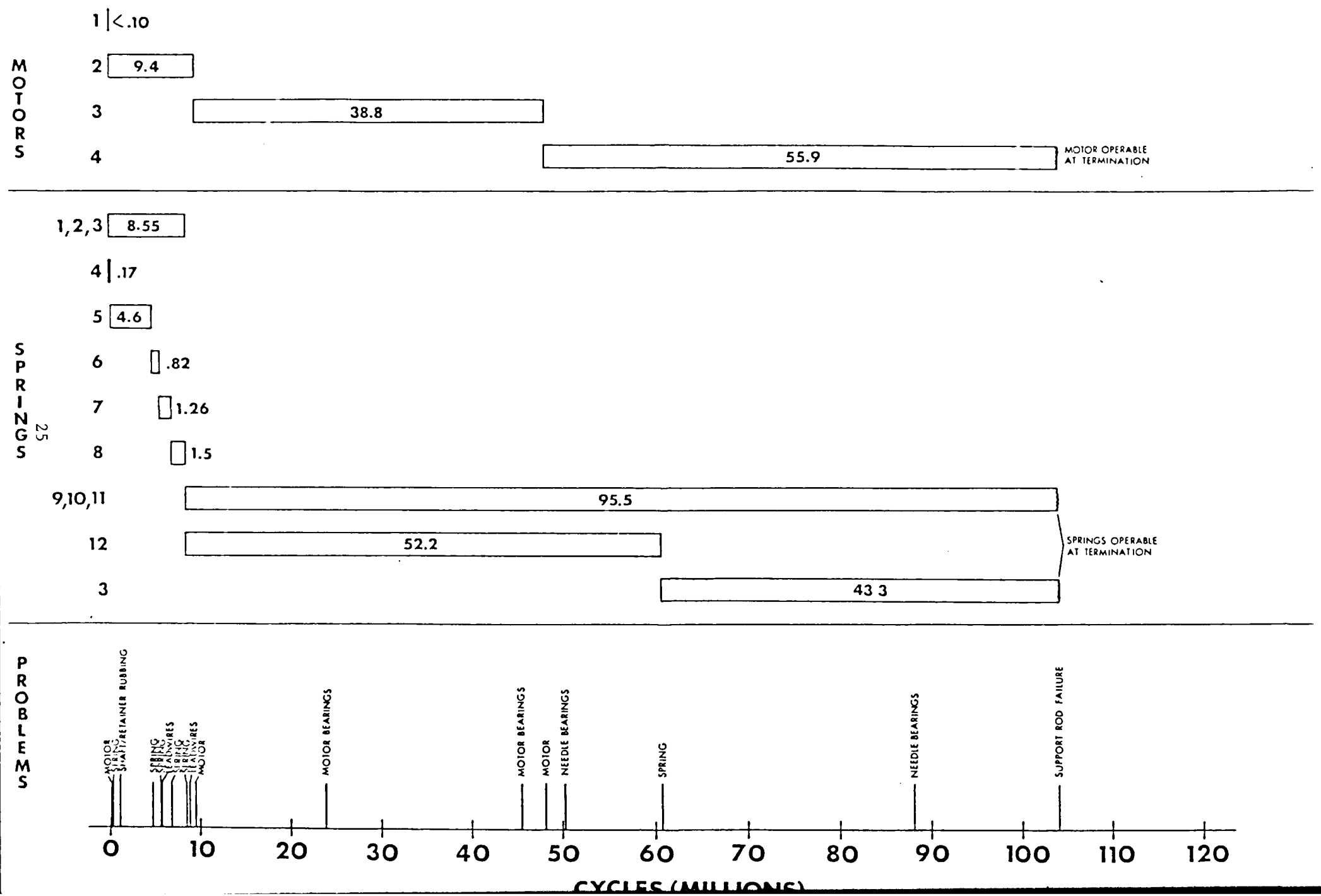
Test Rig Component Life - As with any accelerated life test fixture, the life of the components would determine the feasibility of using the vibration table as an accelerated flex life test rig for film samples. Our experience over the 104 million cycles of operation that the film samples were subjected to has been summarized in the time line of Figure 10. Figure 10 has been divided into three sections with the top two sections devoted to the two major components, the motor and the springs. The bottom portion of Figure 10 is a time line summary of all problems encountered.

Four different motors were used on the table. Within less than 100,000 cycles the first motor failed and we quickly learned that the relatively fine coil wire used in this motor could not withstand the vibration. Each motor design thereafter used increasingly larger diameter wire for the motor windings. This resulted in higher current, lower voltage operation, and with a small decrease in efficiency. However, the results in terms of life of the motor indicated a striking improvement as shown in Figure 10. The second motor lasted 9.4 million cycles, the third lasted 38.9 million cycles, and the fourth was still running at 55.9 million cycles when the experiment was terminated.

The springs were a source of considerable difficulty during the first 10 million cycles of operation. Although three springs lasted 8.55 million cycles and had not broken when they were electively replaced, 5 other springs each lasted only 4.6 million, 1.5 million, 1.26 million, 0.82 million and 0.17 million cycles each during the initial 10 million cycles of operation. After the first spring broke we contacted experts at the supplier company who advised that we increase our preloading of the springs from 0.4 inches to 0.5 inches. The reason for this advice was to be sure to avoid any possible "slapping" of the springs at the peak excursion point of the table, a situation wherein the springs may have been completely unloaded and undergoing impact forces. This suggestion did not appear to help, since 4 other springs broke thereafter. We were also advised that in most applications for the springs the maximum number of cycles to failure was approximately 1 million.

We observed that all but one of these springs failed in the first coil directly opposite the termination point of the spring. We theorized that the termination point was impacting on the next coil, creating a stress rise or stress concentration point. In order to eliminate the problem, cup shaped spring retainers were fabricated and the last coil was press fit into the cups. This modification was made on the frame side of each spring, where all of the springs had been failing. All four springs were replaced with new springs at this point, and three of these springs lasted 95.5 million cycles

FIGURE 10


MOTORS
1 <.10
2 9.4
3 38.8
4 55.9
MOTOR OPERABLE AT TERMINATION
SPRINGS
1,2,3 8.55
4 .17
5 4.6
6 .82
7 1.26
8 1.5
9,10,11 95.5
12 52.2
3 43 3
SPRINGS OPERABLE AT TERMINATION
PROBLEMS
MOTOR
SPRING
SHAFT/RETAINER RUBBING
SPRING
BEARINGS
SPRING
BEARINGS
MOTOR
MOTOR BEARINGS
MOTOR BEARINGS
MOTOR
NEEDLE BEARINGS
SPRING
NEEDLE BEARINGS
SUPPORT ROD FAILURE
0 10 20 30 40 50 60 70 80 90 100 110 120
CYCLES (MILLIONS)

and were operating properly at the termination of the experiment. One of these four springs failed after 52.2 million cycles. This dramatic improvement suggests that the cup shaped spring retainers had eliminated a stress concentration and thereby lowered the peak stress below the fatigue limit of the material.

Although the motor and springs were the source of most of the difficulty maintaining operation of the table, there were a few miscellaneous problems with other features of the vibration table. During the first million cycles we found that the brass spring retainers were rubbing on the support rod shaft, adding too much friction to the operation of the device. These retainers were then attached to the pillow block on one end, and to the locking mechanism on the other end in order to permit the shaft to move freely within the retainers No additional problems were encountered with this design feature.

Motor lead wire failures occurred twice during the first 10 million cycles due to vibration. Although the lead wires were selected because of their multiple strand nature and subsequent ability to withstand significant flexing, the application at first appeared to be too demanding. However, a procedure of leading the wires off the table in the direction of motion of the table and then up to the electronic controller resulted in motion that was more translational than flexural, and the problems with lead wires diminished. This procedure was coupled with that of binding the lead wires together to further dampen vibratory motion. Thereafter lead wires were replaced with each motor replacement. It should be noted, though, that had the motors not been replaced, it would certainly have been necessary to replace the lead wires. Thus, a recommendation for future work is that of replacing lead wires every 40 or 50 million cycles as a maintenance procedure, in addition to taking care to exit the wires in the direction of motion of the table. However, the fact that they proved to last 40 to 50 million cycles indicates an acceptable lifetime for this component.

Additional infrequent problems occurred with motor bearings that needed to be replaced at the 23 million cycle point and at the 45 million cycle point. Motor speed had decreased indicating the presence of an additional load and the cause for the increased load was traced to the bearings. However, the last set functioned for 55.9 million cycles and were operational at the conclusion of the experiment. These bearings were not a source of major difficulty, and in the future could be replaced on a routine basis at 40 or 50 million cycles, and sooner if a drop in speed of operation occurs.

The needle bearings failed twice, indicating that approximately 40 million cycles could be the limit for this component. However, as with the other miscellaneous problems cited above, the replacement is not detrimental to the overall goals of the test fixture and is an readily acceptable maintenance procedure.

C2. Film Sample Preparation Results

In addition to preparing films as described in section B, Foster-Miller subjected film samples to a characterization for tensile properties and orientation angle. Except for minor editing, the results in this section are as reported to Nu-Tech by Lucy Elandjian of Foster-Miller in her final report.

Table 1 describes the tensile properties of the heart diaphragms materials. The "volume percent of PBZT" refers to the percentage of PBZT in the whole composite, which includes polymer (especially in the case of polyurethane) on the surfaces of the film.

The results of the mechanical tests confirm that the IPN composite fails in a ductile manner with a well-defined yield point and over 7 percent elongation for thin and over 25 percent for thick PBZT at break. Figure 11 shows the stress-to-strain relationship of a thin and thick neat PBZT, and thin and thick PBZT/Polyurethane composite and a polyurethane film. The IPN composite has the best of both materials: the high strength of PBZT and the low modulus and high elongation of polyurethane. This combination makes it an ideal high toughness material for heart diaphragm application. This should be reflected by excellent fatigue life for the PBZT/polyurethane composite film.

In conclusion, these results prompt Nu-Tech and Foster-Miller to be optimistic that polyurethane PBZT composite films will be a significant improvement in the development of artificial heart diaphragms. The goal of delivering $\geq$ 5 year in-service life expectancy is within reach of this composite material as it combines the high tensile strength of PBZT with the toughness and flexibility of polyurethane.

C3. Biocompatibility Test Results

Physicochemical Tests - The conclusion drawn by NAMSA for Both the PBZT and PBZT IPN materials was that USP limits were met. Specifically, both materials were found to have identical results as noted below:

	PBZT	PBZT IPN	USP Limits
Non-Volatile Residue	1 mg	1 mg	15 mg
Residue on Ignition	$\leq$ 1 mg	$\leq$ 1 mg	5 mg
Heavy metals	< 1 ppm	< 1 ppm	1 ppm
Buffering Capacity	< 1 ml	< 1 ml	10 ml

Cytotoxicity - MEM Elution - The following identical results were reported by NAMSA for each of the test samples submitted to this test.

	Confluent Monolayer	Intracellular Granulation	Swelling	Crenation	% Lysis	CTE Score
24 HOURS						
Test Extract	(+)	(-)	(-)	(-)	0	N
Negative Control	(+)	(-)	(-)	(-)	0	N
48 HOURS						
Test Extract	(+)	(-)	(-)	(-)	0	N
Negative Control	(+)	(-)	(-)	(-)	0	N
72 HOURS						
Test Extract	(+)	(-)	(-)	(-)	0	N
Negative Control	(+)	(-)	(-)	(-)	0	N

Table 1. PBZT Tensile Properties

	Thin, Neat	Thin, IPN (poly-urethane)	Thick, Neat	Thick, IPN (poly-urethane)	Thin IPN (silicone)
Thickness, (in.)	0.0002	0.0013	0.0012	0.0033	0.0004
Volume % of PBZT	100%	15%	100%	36%	50%
Tensile Strength (ksi)	66	20.2	43.8	20.3	15.2
Tensile Modulus (Msi)	3.96	0.98	3.92	0.65	1.67
Elongation	1.8%	7.2%	1.1%	25.7%	1.4%

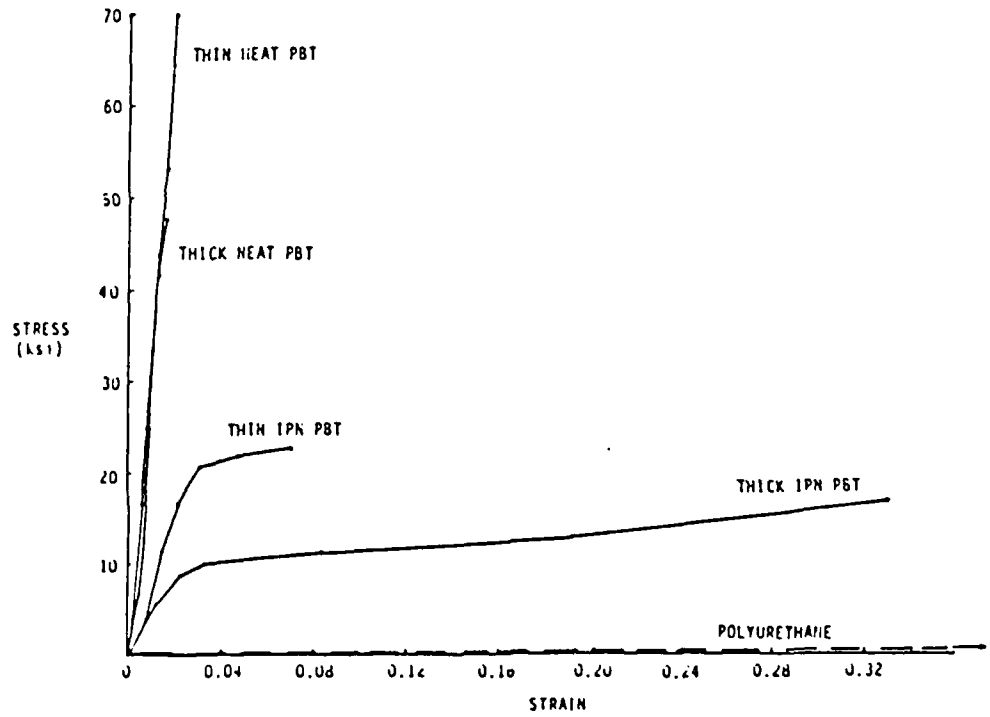


Figure 11. Stress-Strain Relationship of PBZT, Polyurethane and PBZT/Polyurethane Composite Materials

Positive control, T-6500-5-2, was toxic at a dilution of 1:2 at 24 hours.

Conclusion: Nontoxic

Hemolysis Test (In vitro)

The results of the hemolysis test for the PBZT are as follows:

Negative Control = 0.04 absorbance = 0 % hemolysis
Positive Control = 2.0 absorbance = 100 % hemolysis

Test #1 = 0.04 absorbance = 0 % hemolysis
Test #2 = 0.04 absorbance = 0 % hemolysis

Mean Hemolysis 0 %

Under the conditions of this test, the material would be considered nonhemolytic.

The results of the hemolysis test for the PBZT IPN material are as follows:

Negative Control = 0.04 absorbance = 0 % hemolysis
Positive Control = 2.0 absorbance = 100 % hemolysis

Test #1 = 0.05 absorbance = 1 % hemolysis
Test #2 = 0.05 absorbance = 1 % hemolysis

Mean Hemolysis 1

Under the conditions of this test, the material would be considered nonhemolytic.

Muscle Implantation Test - Results of this test for both the PBZT and and PBZT IPN materials yielded the same conclusions. Macroscopically neither material resulted in the formation of a tissue capsule, and thus with respect to the pathology scores, there was no difference noticeable in comparison with the control. Specifically, the pathology report stated: "Macroscopic: The reaction was not significant as compared to the negative control implant material." In terms of the microscopic, the pathologist scored both materials as: "Microscopic: The reaction was a slight irritant as compared to the negative control implant material."

Discussion with the pathologist indicated that in light of the above three additional tests, all of which showed excellent results, and in light of the fact that the implant did not show any signs of tissue necrosis, the conclusion of a "slight irritant" should not cause any concern at this point. Furthermore, the "slight irritant" conclusion, without tissue necrosis, led the pathologist to conclude that the implant was merely indicating the possibility of a foreign body reaction, or a "walling off" of the material. This type of reaction is known to exist with current artificial hearts materials, and thus this conclusion is further indication that there is no particular concern over the "slight irritant" conclusion. Future testing, however, as recommended by the pathologist, would include a much longer implant duration of, for example, 90 days.

As a result of all four tests, and as a result of discussions with the pathologist, we feel that a conclusion that "biocompatible feasibility" has been demonstrated is warranted. This conclusion is drawn while recognizing, however, that more strenuous tests should be performed, such as those relating to a determination of the potential thrombogenicity of the material.

C4. Film Sample Flexural Fatigue Test Results

Samples were examined at 48.2 million, 87.1 million, and 104.1 million cycles, under 20 power with a stereomicroscope. The same individual performed all of the examinations so that there would be consistency in the reporting. The observations at 48.2 million and at 104.1 million cycles have been summarized below in some detail, as a means of presenting the basic information. Conclusions that could be drawn are made at the end of this section.

Observations at 48.2 million cycles

The best means to present summary information for this point in the test was a general description broken down by sample type.

Thin PBZT/Silicone - This material showed consistent patterns of wear and fatigue. Samples exhibited typically four distinct zones or patterns. At each end, slightly above the clamp area, zones of green blotches appear. The surface silicone material gave the appearance of flaking off, leaving fibers coming up out of the parent material. The edges at these sites show significant deterioration, with a "V" shaped wedge of missing material working its way inward. The other two zones typically were found on either side of the center of the specimen. These zones were found to have numerous small cracks and many fibers sticking up from the surface giving it a furry appearance.

Thin PBZT - This material was very much like the Thin PBZT/Silicone above. Its zonal characteristics were almost identical. In the opinion of the examiner, this material seemed to withstand the flexure slightly better as judged by the number of remarkable features noted.

Thin PBZT/Vibrathane - Overall, this material withstood the cycling flexure very well. However, there were some areas of fibrous material and in one case a large section had been torn from the sample, which the examiner attributed to poor sample preparation as opposed to fatigue failure.

Thick PBZT - This material, of comparable thickness to the Thin PBZT/Vibrathane (and thus markedly different in thickness from the first two materials listed above), withstood flexing very well. Typically, the observation was that fibers were present at the edges, although there were only a few fibers coming from the surface.

Thick PBZT/Vibrathane - Similar findings to the Thick PBZT above.

In general, at this point it appeared that the trend was for the thicker materials to withstand the flexing better. However, since the thin materials exhibited somewhat more extensive motion (i.e. secondary nodal points of vibration or flexure), it is possible that the thicker material held up better

simply because the fixture did not cause the same degree of motion. The green appearance could have been caused by lubricants from the bearings leaking down onto the samples. Future testing should utilize a lightweight enclosure to eliminate such an effect and to generally keep the samples cleaner.

Observations at 104.1 million cycles -

A more elaborate recording of the summary information is presented at this, the last observation, in order to provide the reader with a more in depth appreciation of the findings. In the following listing the observations for each sample have been recorded. At the end of this section trends and conclusions will be discussed.

Thin PBZT -

Sample 1) Many wear holes in the material, specifically at high stress areas and edges. Also at high stress areas there were a great deal of fibers lifting from the surface.

Sample 2) Wear holes, possible due to rubbing against the Angioflex material. Triangular shaped tears at high stress zone giving the appearance of the material delaminating.

Sample 3) Material failed at the clamp. Wear holes present. Reason for failure is questionable, however, and could possibly have occurred during maintenance of the vibration table.

Sample 4) Questionable failed sample. Previous inspection had noted a tear forming at one clamp, the same end which tore free. However, this could possibly have occurred during maintenance of the table.

Sample 5) Sample missing. Previous inspection had shown a large tear forming at one end with the tear forming from wear holes in the material. This piece had been on a sample block that had fallen off when the support rod failed.

Sample 6) Sample had an elongated wear hole parallel with the edge, possible due to wear against the Angioflex. Some fibers were found to be lifting from the surface and a dark green, almost black zone was found at the high stress area, but nothing as severe as found on previous samples.

Sample 7) This sample had severe decay of the edge, due possibly from wear with the Angioflex. The holes parallel with the edge were seen in an earlier inspection, and likely led to the decay and missing segment of material.

Sample 8) A small piece missing at the edge of the zone of high stress with elongated holes due to possible wear with the Angioflex. This sample was not as severely damaged as others.

Sample 9) This sample has a piece missing at the edge in the high stress zone, a series of elongated holes along one side, another triangular tear (with the appearance of delamination) at the stress zone, and two other zones of green with fibers erupting from the surface.

Thin PBZT, general remarks:

This was one of the materials that was so thin that it required the underlayment material so that it could be tested. The material itself had so much static electricity that it would stick to itself and so light that it would not move in a consistent fashion. With the Angioflex as underlayment and the amount of dirt and debris that the fixture deposited on the samples, the Angioflex could have worked like sandpaper on the PBZT. A consistent observation was that fibers would erupt from the surface in the areas of high stress, and these fibers were almost certainly due to flexure and not wear. Some tears that occurred at the edges appeared to be a delaminating of the material itself, i.e. a tear would start but would peel apart as it propagated. Wear holes along sides and at high stress points near the clamp regions appeared to be due more to the wear against the underlayment material.

Thick PBZT -

Sample 1) The only noteworthy change is a few fibers coming out of the surface. The fibers along the edge were present, to a lesser extent, from the start of the testing.

Sample 2) A small tear was found at the clamp, of questionable origin. A few fibers were found erupting from the surface and along the edge.

Sample 3) Fuzzy, fibrous edges with a few places on the surface where fibers erupted into ball shapes. There was a zone of green above the clamp that had fibers lifting up from the surface.

Sample 4) Fibers found along one edge. A small tear of questionable origin found on one edge and a fold of material above the clamp that took on a greenish tinge.

Sample 5) When the sample holder failed due to the failure of the support rod, the sample was torn. In addition, the edges near a missing segment were jagged and had a greenish cast to them. Otherwise, the piece did not exhibit any significant signs of deterioration.

Sample 6) Nothing remarkable except for a band of green in a high stress area and a small hole. The hole appeared to be worn, but the cause was unknown.

Sample 7) Nothing notable beyond a few fibers at the edge and a couple of the surface.

Sample 8) Nothing notable except a few fibers forming circles on the surface

Sample 9) Fuzzy, fibrous edges, circles with fibers at the center, and a green spot with some fibers.

Thick PBZT, general remarks:

The deterioration in these samples was primarily exhibited by eruption of fibers from the surface, although the deterioration was not great in comparison to the thin materials. While fibrous edges were also noted, these could have

caused during the cutting of the samples. The edges looked fractured.

Thin PBZT/Vibrathane -

Sample 1) Except for some fibers at the edge, little deterioration was exhibited. In fact, small pin holes noted prior to testing did not grow.

Sample 2) Except for light green areas at high stress areas above the clamps, nothing notable.

Sample 3) Some fibers lifting from the surface (noted in previous examination) and one zone above the clamp in the high stress area that had numerous cracks running laterally across the surface.

Sample 4) A small tear in the side of questionable origin and a green zone above the clamp in the high stress region.

Sample 5) Other than an elongated piece missing from the previous inspection and a few zones of where the coloration appeared lighter (above the clamp areas), the material exhibited little deterioration. The clamp on this sample bar failed when the support rod failed, and tore the material in two. The piece noted missing originated in a jagged cut region of the material.

Sample 6) This material was found to be torn in two during the previous inspection. The cause of the tear was unknown, but the sample was very dirty and oily. The area where this occurred was green and jagged in appearance.

Sample 7) Nothing notable except for a dark band above the clamp.

Sample 8) Nothing notable.

Sample 9) Except for a small tear at the side, noted at the 48 million cycle mark, which did not propagate, there was nothing really notable.

Thin PBZT/Vibrathane, general remarks -

Of the thin materials this appeared to withstand flexure the best, although it must be noted that we refer to the material as "thin" to differentiate it from another PBZT/Vibrathane material that was 3.3 mil thick. This "Thin" PBZT/Vibrathane was actually 1.3 mil, which was very close in thickness to the "Thick" PBZT material that was 1.2 mil thick. Relatively few fibers were found erupting from the surface.

Thick PBZT/Vibrathane -

Sample 1) Small tear at clamp noted in previous inspection, but the tear did not propagate.

Sample 2) Small tear at clamp noted at the 48 million cycle mark that tore completely.

Sample 3) Fiber at the edge and a few fibers on the surface.

Sample 4) A few fibers in one area, but otherwise nothing remarkable.

Sample 5) A few fibers visible on the edge, otherwise nothing remarkable.

Sample 6) Clamp failure completely removed the center of the material. A few fibers were noted, but otherwise nothing remarkable.

Sample 7) Nothing remarkable.

Sample 8) A few striations above the clamp, but nothing else remarkable except that the sample was found to be particularly dirty.

Sample 9) A few fibers found with circles around them, but nothing else remarkable except that the sample was found to be particularly dirty.

Thick PBZT/Vibrathane, general remarks -

Other than a few fibers, the damage to the samples appeared to have occurred due to failure of the structural components of the vibrating table and sample holder. The material samples appeared to be very durable. However, this material was particularly thick, and therefore may not have moved to the same degree as other samples.

Thin PBZT/Silicone -

Sample 1) Material appeared to have four high stress zones, apparently linked to secondary nodes of vibration. All four showed a great many fibers coming up from the surface. One of the high stress zones, located near the clamp and thus at a region of greatest flexure and possibly greatest wear against the Vibrathane exhibited numerous holes.

Sample 2) Zones of fibers lifting up, edge wear due to the underlayment material, small tears with delamination and fibers.

Sample 3) At the previous inspection, a large section was noted to be missing with a piece of the missing section adhering to the angioflex. Cause suspected to be friction and wear during motion against the Vibrathane.

Sample 4) Edge holes forming from wear with Angioflex. Sample was torn by the sample clamp failure, which also left tears and folds in the material.

Sample 5) Sample clamp failure destroyed this piece also. Material had curled up on the clamp. Deterioration of the sample was difficult to differentiate between fatigue and damage due to the test rig failure.

Sample 6) Three zones or areas of fibers were found lifting out from the surface and one of these zones appeared to have many cracks running laterally across the material.

Sample 7) One green zone was found at the high stress area that had a piece missing at the edge and with a hole wearing through. A small tear was found to be forming at a point where fibers were erupting from the surface. This also had the appearance of delaminating material.

Sample 8) Three zones of green were found with many fibers lifting out of the material. One zone above the clamp area had a string of holes across the material that appeared to be due to wear and not flexural fatigue.

Sample 9) Material seemed to be failing from the edges. Green zones with cracks and fibers that resulted in small tears or delamination of the material were also observed.

Thin PBZT/Silicone, general remarks -

This material did not withstand the cycling well at all. The mechanics of its failure appear to be related to zones where cracking is observed, followed by fibers erupting from the surface, with tears and the appearance of delamination.

Angioflex -

None of the samples showed any deterioration, except for those damaged by the sample bar clamp failure. Some of the samples took on a coloration of the PBZT. Otherwise, nothing remarkable was noted.

Fatigue Test Discussion and Conclusions -

The best comparisons between material types are drawn when materials of similar thickness are compared. In comparing the Thin PBZT/Silicone with the Thin PBZT the general finding was that neither material withstood the flexing. Eruption of fibers from the surface was the predominant finding, which we feel strongly was related to fatigue due to the location within the high flex regions. The silicone thus did not appear to improve the basic flexural capability of the PBZT. The examiner's conclusion was that if anything, there was a possibility that the IPN material actually was exhibiting more deterioration with fatigue than the PBZT neat. However, conclusions are particularly difficult to draw with these thin materials, because the thin materials exhibited far more pin holes with increasing cycle time, which we feel were likely to be more related to wear than to fatigue.

Perhaps the best comparison between materials was found in comparing the Thick PBZT (1.2 mil) with the Thin PBZT/Vibrathane (1.3 mil). The thickness was close enough that it was doubtful that thickness would have significantly influenced the results. The primary difference between these materials was found in terms of the number of fibers erupting from the surface. The IPN material had only one sample reported with this failure mode, while the Thick PBZT exhibited six of the samples with this feature noted. As noted above, the eruption of fibers in other material samples appeared at times to be associated with cracks. After the fibers appeared the material had a tendency to tear or delaminate from this site. Since the fibers were located in regions of high flexural motion, these observations lead us to conclude that the appearance of the fibers was due to flexural fatigue. Because of the observations of far fewer fibers erupting from the surface in the Thin PBZT/Vibrathane material, in comparison with the Thick PBZT material, the results suggest that the process of forming an IPN with Vibrathane improved the basic fatigue properties of the PBZT.

Since the Angioflex material held up very well, and the PBZT/Vibrathane materials showed indications of failure, clearly the samples of PBZT/Vibrathane do no yet have the quality needed to replace the polyurethaneureas. However, when one considers that the processing techniques for the PBZT materials have not yet been refined to the same degree as the Angioflex material, there is still a possibility that the PBZT materials would have the potential to be improved sufficiently. Our observations of the material at Nu-Tech lead us to comment on what we feel was a striking lack of homogeneity in the materials, from a visual standpoint. We recognize that the structure of the materials, both the PBZT neat and the PBZT IPN is such that homogeneity should exist to a fine scale. However, the macroscopic appearance of the materials does not provide a homogeneous appearance. Furthermore, the eruption of fibers as a key feature in the failure mode raises questions also about the homogeneity. Perhaps improvements in processing will eliminate these problems, and if they are eliminated then we might see the long flex life that we have expected.

The numerous holes found in the thin samples were most likely due to wear. The tendency for friction between these samples and the Angioflex underlayment material to wear holes in the thin PBZT materials was probably increased by the dirt that adhered to the samples, i.e. there may have been an additional abrasive substance introduced.

Although the Angioflex material did not fail during the 104 million cycles of the test, due to the fact that the PBZT materials failed, and failed apparently from fatigue, we conclude that the test fixture provided sufficient flexural stress to warrant continuing investigations into its use as a flexural fatigue tester. Although the test rig design as it existed at the end of the experiment provided adequate test time between component failures and/or maintenance to justify its use as an accelerated life test fixture, the potential for improvements discussed in the next section could make the device even more attractive.

D) RECOMMENDATIONS FOR FUTURE WORK

The accelerated life test fixture, in its final form at the end of this development, provided a means to obtain flexural testing within a couple of months as opposed to years of real time testing. However, we have identified several areas of improvement in future designs.

Excessive friction on the table that could most likely be reduced would be at the location where the springs contact a brass retainer. Friction was found to be high enough to continually produce brass particles. Some type of bearing arrangement at the contact between springs and the retainer might reduce the friction considerably. This reduction in friction would impact the overall design in a favorable fashion from the standpoint of requiring less motor torque. This would in turn permit the table to be operated at larger amplitudes and/or higher rates.

In the next design it would be appropriate to expend some design effort on a lightweight enclosure for the test samples. The present design exposed the samples to dirt and lubrication that was shed from the bearings.

Yet another design change that could be worth exploring would be the use of a flexible drive cable, thereby permitting the motor and its mass to be removed from the table. Although the final motor design used in this study performed

remarkably well in the face of significant vibration, removal of mass from the table would improve performance considerably. The drive cable would have to be brought off the table in the direction of motion of the table, much like the lead wires to the motor were brought off the table in the present design, in order to minimize flexing of the cable.

In terms of testing the materials, we would recommend confining the testing to materials of the same thickness. Experimentation with varying lengths of the samples could prove to be desirable as a means to increase the motion. Use of the Angioflex material as an underlayment appeared to be a good method of forcing the PBZT materials to move in a consistent fashion, and has an advantage of providing a control for comparison for each PBZT sample. The disadvantage was the wear exhibited by the thin samples, and thus we would recommend that the samples be at least 1 mil in thickness.

An investigation from a materials processing standpoint appears warranted in order to determine the nature of the fibers that were found to erupt from the materials surface as the fatigue failure mechanism. If the material processing can be improved, there is a significant opportunity to provide a high flex life material from the PBZT. Furthermore, if the prepolymer of a polyurethaneurea could be obtained instead of the Vibrathane, results might significantly improve.

Biocompatibility testing should include longer duration muscle implantation testing to assure that there are no long term toxic effects. Furthermore, testing with respect to the potential for the material to be thrombogenic should be pursued as part of the next level of biocompatibility testing.

Perhaps the best potential for ordered polymer films to provide a high flex life material for biomedical applications comes from other ordered polymers in the same family. Nu-Tech Industries is aware that private industry is working on such a material that would be more flexible than the PBZT materials tested in this study. Ordered polymers of the family used in this study may hold the key to high flex life materials for biomedical and aerospace applications.

E. LITERATURE CITED

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